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MALFUNCTION INVESTIGATION AND CHEMICAL ANALYSIS CAPABILITY MANUAL

Prepared by
MALFUNCTION ANALYSIS BRANCH
ANALYTICAL LABORATORIES DIVISION

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INTRODUCTION

This handbook describes the capabilities of the Malfunction Analysis Branch (MAB) and the equipment available, and outlines the methods by which requesters can best employ the MAB services.

The handbook explains the operations of the branch, its mission, the services available, and its capabilities. A concise description is given of the equipment and facilities utilized to conduct investigations, analyses, and tests.

The MAB consists of a team of postfailure investigative specialists and their supporting equipment. To meet launch site requirements for services, the scope of specializations and equipment capabilities is necessarily quite comprehensive.

MAB services are available to all NASA-Kennedy Space Center (KSC) organizations, NASA contractors, and other government agencies operating at KSC.

Services of a problem-solving nature may require the use of several different professional specialists and instruments in an unpredictable sequence. The MAB personnel are trained to determine the needed processing and to perform and/or direct the work through the various laboratory functions. If the requester does not know specifically what his task requires, he will benefit by presenting the overall problem to the MAB, rather than a request for a specific function.

Problem-related laboratory work should be presented to the Malfunction Investigation Laboratory (MIL) supervisor through the requester's authorized single point of contact. The information flow, priority, reporting process, and other factors will be resolved by these cognizant personnel.

SECTION I
ORGANIZATION

1.1 GENERAL

The MAB consists of two operating sections. The sections are designated:

a. Malfunction Investigation Laboratory Section

b. Support Laboratory Section

The laboratories within each section, as a composite group, have full capabilities for the malfunction investigation, physical testing, and chemical analysis as described in this document.

Each laboratory contains its own specialized equipment and instruments. However, tests and investigations often cross laboratory boundaries, and branch personnel are cross-trained in the principles of operation and capabilities of instruments. Cross-training among specialization enables an investigator to know when to seek the counsel of other specialists.

The MAB is committed to respond to emergency situations and, where justifiable, its services will be made available at any time. During normal working hours, calls regarding malfunction analysis should be made directly to the Malfunction Investigation Laboratory Section (867-7048, 7049, or 7050), Room 1233 of the Manned Spacecraft Operations Building, M7-355 (MSOB), sometimes known as the Operations and Checkout (O&C) Building. Calls regarding nonroutine chemical analysis should be made directly to the Support Laboratory Section (867-6591, 6592), Room 1274 of the MSOB.

1.2 MALFUNCTION INVESTIGATION LABORATORY SECTION

The Malfunction Investigation Laboratory Section is the MAB entry point for all failed parts and for problems needing analysis and investigative work.

Interface problems that may have caused a failure or malfunction are investigated and analyzed by the laboratory prior to any disassembly operation. Nondestructive techniques will be used to study parts that are hidden from view inside assemblies, or potted components, prior to any disassembly or destructive testing.

Nationwide liaison is maintained with other government agencies and with industry to ensure that up-to-date testing techniques and information relative to components and materials in components are available.

1.3 SUPPORT LABORATORY SECTION

The Support Laboratory Section analyzes and identifies materials and their chemical and physical characteristics to determine the specific causes of failures, and performs general nonroutine chemical analysis.

The Support Laboratory Section is staffed and equipped to conduct qualitative and quantitative chemical analysis of materials and compounds. The equipment ranges from that used for wet chemical procedures to that used on samples tens to thousands of times smaller than is possible with wet chemical procedures.

The laboratory is separated into two functional areas; however, the interface in the overall scope of the mission is so close that an inseparable composite is formed. The functional areas are:

a. Analytical Instrumentation

b. Wet Methods

The support equipment used includes equipment necessary for the preparation of samples for microscopic and analytical examination; and for various spectra files, X-ray powder data files, and comparison standards.

SECTION II
FACILITIES AND EQUIPMENT
MALFUNCTION INVESTIGATION LABORATORY SECTION

2.1 STAFFING

The Malfunction Investigation Laboratory (MIL) staff specialties include mechanical and electronic engineering, physics, chemistry, materials and metallurgy, metrology, and photography.

Services of a problem-solving nature may require the use of several different specialists and instruments in an unpredictable sequence. The MIL personnel are trained and qualified in thorough investigative techniques, documentation, and written presentation of findings. They determine the required tests and evaluations which must be accomplished in any given investigation, determine the optimum test sequence, conduct the evaluation and/or expedite the material or component through the various laboratory functions, reduce the data and make the necessary scientific deductions, research the problem for recommended corrective measures, and present findings in a technical report.

2.2 FACILITIES

The Malfunction Investigation Laboratory is composed of three main laboratories: (1) the Electronics Laboratory, (2) the Metallurgical Laboratory, and (3) the Mechanical Systems and Fluids Laboratory.

2.2.1 ELECTRONICS LABORATORY

2.2.1.1 Present Capabilities. The electronics laboratory performs investigative evaluation of electrical and electronic subsystems, circuits, and components. The laboratory is equipped with a broad selection of high-quality test instrumentation for measurement of basic parameters in electrical systems to confirm, isolate, and identify the basic failure mechanisms and the initial cause of failure. Systems, circuits, and components are examined and evaluated as to their design, application, and manufacturing

techniques. Subsystems and modules are dissected as necessary to determine the initial point of failure. Individual components may be sectioned, as necessary, to determine the failure mechanism and its cause. Microcircuits and semiconductors may be sectioned and incrementally polished through their junctions. Optical micrographs, scanning electron micrographs, and infrared micrographs of junctions, junction failures, and other circuit component failures are prepared as required to isolate and identify basic failure mechanisms.

The present laboratory, by design, was restricted to low- and medium-power capabilities, and maximum frequency capabilities of approximately 30 MHz. All basic electrical parameters can be accurately observed within the above limitations.

2.2.1.2 Future Capabilities. Present and anticipated support requirements have dictated that the laboratory extend its capability to include high-frequency parameters. The laboratory is currently developing a capability to measure all network parameters including: phase, gain, attenuation, impedance, return loss, reflection coefficients, and S parameters, for components and circuits, over the frequency range to 12.4 GHz. The system can easily be extended to 18 GHz if required. It is anticipated that a complete high-frequency capability will be realized during Fiscal Year 1971.

2.2.2 METALLURGICAL LABORATORY

The metallographic analyses performed in the metallurgical laboratory of the MIL consist of studies of fracture surfaces through low- and high-power light optics in an endeavor to pinpoint failure origins. Metallographic studies of the microstructure, supplemented by scanning electron micrographic studies of the origin of failure, identify modes of failure and/or material defects. In addition, the metallographic studies identify anomalies within the microstructure produced through improper manufacturing techniques, as well as identifying improper alloy selection or substitutions. Hardness and microhardness determinations aid in identifying improper heat treatment and adverse phase changes within the microstructure, respectively. Metallurgical analysis of failed components enables an objective review to

be made which normally leads to recommendations of design and/or material changes for function, safety, and economic considerations. Protective coatings and platings are also recommended where necessary for corrosion prevention.

2.2.3 MECHANICAL AND FLUIDS LABORATORY

The mechanical and fluids laboratory is staffed and equipped to perform malfunction investigative work on all KSC mechanical hardware, including pneumatic and hydraulic static and dynamic fluid flow problems.

Every attempt is made to determine, isolate, and evaluate the primary step in any problem causality train, so that the most basic problem area causes may be identified and eliminated.

2.3 EQUIPMENT

2.3.1 ELECTRONICS LABORATORY

2.3.1.1 DC Power Supplies. Numerous dc power supplies are on hand for electronic testing. Most are the variable-voltage-regulated type. In total, they are capable of covering a voltage range of 0 to 500 vdc, 0 to 125 amps dc. Several are the variable-voltage-regulated, current-regulated type, covering a range of 0 to 150 vdc, and 0 to 15 amps dc.

2.3.1.2 Signal Sources

- Secondary DC Voltage Standard, Hewlett-Packard Model 740B
- Secondary Frequency Standard; output frequency standards are available from the Hewlett-Packard Model 5256L Electronic Counter
- General Radio Type 1390B Random Noise Generator
- General Radio Type 1311-A Audio Oscillator, Variable 380 to 420 Hz, 20 to 150 VAC Sine Wave
- Hewlett-Packard Model 200 CD Wide Range Audio Oscillator with range from 0 to 600 kHz
- Hewlett-Packard Model 652A Test Oscillator, 0 to 10 MHz
- Hewlett-Packard 3300A Function Generator
- Hewlett-Packard Model 214A Pulse Generator, 0 to 1 MHz

2.3.1.3 Metrological Instruments

- Hewlett Packard 5245L Electronic Counter, with 5267A Time Interval Unit and 5257A Transfer Oscillator Unit, which measures time intervals from 1 microsecond to 10^8 seconds and frequencies to 18 GHz
- Hewlett Packard 740B Differential Voltmeter
- Dymec 2401A Integrating Digital Voltmeter
- Systron 1031-7 Counter Timer
- General Radio 1615-A Capacitance Bridge
- General Radio 1608-A Impedance Bridge
- General Radio 1311-A Audio Oscillator

2.3.1.3 Metrological Instruments (Continued)

- General Radio 1232-A Tuned Amplifier and Null Detector
- General Radio 1142-A Frequency Meter and Discriminator
- General Radio 1644A Megohm Bridge
- Tektronix Type 575 Transistor Curve Tracer Oscilloscope
- Tektronix Type 549-A Storage Oscilloscope, with 1A1 Dual Trace; and 1A7 and 1A5 Differential Amplifier Units; 1S1 Sampler Unit; and 1L5 and 1L10 Spectrum Analyzer Units
- Hewlett Packard 3440A Digital Voltmeters (5) with AC, DC, and Multifunction Plug-In Units
- Hewlett Packard 7702A Dual Channel Oscillographic Recorder with 8803A High Gain Preamplifier
- Hewlett Packard 334A Distortion Analyzer
- Hewlett Packard 410C Electronic Voltmeter
- CMC Model 225B Universal Counter-Timer
- Hewlett Packard 457A AC-DC Converter
- Hewlett Packard 6824A DC Power Supply Amplifiers (2)
- Dymec 2410A AC-Ohm Converter
- Simpson 1701 Multi-Range DC Ammeter Standard

2.3.1.4 General Purpose Instrumentation

- Triplett Table Top Transistor Test Set
- RF Detector Probe

2.3.1.4 General Purpose Instrumentation (Continued)

- Load Bank, 300 Ampere, 28 VDC
- Variable Band-Pass Filters
- Decade Resistors, Capacitors, and Inductors
- Volt-Ohm General Purpose Meters

2.3.1.5 Infrared Microscope. The Infrared Microscope (figure 2-1) provides accurate temperature measurements of small target areas. It features direct temperature readout, and the user can view the target while the measurements are in progress. The target area under investigation is not touched during the measurement. Consequently, the microscope is ideal for nondestructive testing of delicate objects, and for thermal measurements of moving or rotating parts. Many kinds of targets can be accommodated by a choice of available substages and objective lenses. For special applications, the optical head can easily be removed for separate vertical or horizontal mounting.

The Infrared Microscope is composed of an optical head, an electronic control unit, and interconnecting and power cables. The optical head includes a stand and post, a Manual Substage having 1-inch by 1-inch travel and calibratec micrometer screws, and a 15X Objective Lens. A 74X and 52X lens are also available when required and the optical eye piece has a 10X magnification. The detector is the type utilizing indium antimonide mounted in an aluminum-jacketed dewar. The measurable temperature range is from 15° C to 165° C, with a system bandwidth of 1 Hz to 400 Hz. Minimum spot size observable is 0.0014 inch with the 15X Objective Lens.

2.3.2 METALLURGICAL LABORATORY

2.3.2.1 Vickers 55 Metallograph. The Vickers 55 Metallograph (figure 2-2) is equipped with transmitted and incident illuminators for macro and micro examination, depending on the area of interest of the specimen. Transmitted polarized light, incident

polarized light, incident dark-field illumination, and phase contrast are available accessories to the Vickers 55 Metallograph. In addition, a pneumatic microhardness testing accessory, and an automatic integrating and manual photographic capability for 35mm and polaroid pictures are available. The instrument is also used for biological and petrographic studies.

2.3.2.2 Leitz Wetzler "Aristophot" Macro Camera. The Leitz Wetzler "Aristophot" Macro Camera (figure 2-2) supplements the metallographs by documenting the object 3/4X to approximately 30X. It is equipped with numerous lenses and illuminators.

2.3.2.3 Leitz Wetzler "Panphot" Metallograph. The Leitz Wetzler "Panphot" Metallograph (figure 2-2) is equipped with transmitted and incident illuminators for macro and micro examination depending on area of interest. The metallograph contains accessories for transmitted and incident polarized light, incident dark-field illumination and incident phase contrast. A micro and macro interferometry accessory is also available. The metallograph is equipped with 35mm and polaroid photographic accessories.

2.3.2.4 Microscopes. Several low-power binocular microscopes with the necessary illuminators are available throughout the laboratory. These microscopes are used for preliminary studies ranging from 7X to approximately 80X.

2.3.2.5 Cut-Off Saw. The laboratory is equipped with an automatic abrasive cut-off saw (figure 2-3) for precision cuts in removing areas of prime interest from failed items. In addition, the unit, equipped with a stationary or rotating vice, is used in the fabrication of fixtures and jigs, as necessary, during the course of a failure analysis.

2.3.2.6 Tukon Microhardness Tester. The Tukon Microhardness Tester (figure 2-4) uses both the Knoop and 136 Diamond Pyramid Indentors with loads from 1 gram to 1,000 grams. The instrument can be used to determine hardnesses of micro-constituents or individual phases within the microstructure; it is an extremely accurate instrument to aid in failure analyses.

2.3.2.7 Electrolytic Etcher/Polisher. One of several electrolytic etcher/polishers (figure 2-5) available in the laboratory is used in the final step of preparation of a metallographic specimen to reveal its microstructure.

2.3.2.8 Ultrasonic Cleaner. An ultrasonic cleaner (figure 2-5) is available to ensure that the specimens, during sample preparation, will remain free of particulate contamination.

2.3.2.9 Automatic Vibrating Polisher. An automatic vibrating semifinal and final polishing apparatus (figure 2-5) is available which allows the investigator to perform other tasks while sample preparation is continued automatically.

2.3.2.10 Heat Treating Furnaces. The laboratory contains various heat treating furnaces (figure 2-6) with capabilities of supplying inert atmospheres when necessary. Included is a vacuum furnace, 2,500° F muffle and crucible furnaces, and several smaller special application furnaces.

2.3.2.11 Lapping Wheels. Six manual and automatic lapping wheels, similar to those shown in figure 2-7, are available in the laboratory. An adequate supply of the special cloths and abrasives to meet any metallographic demand is maintained. A supply of wheels of various sizes and alloys is also maintained.

2.3.2.12 Vacuum Cathodic Etcher. A vacuum cathodic etcher (figure 2-8) is available to facilitate metal composites, refractory alloys, and cermet etching for microscopic examination. It is a special application instrument adapted for everyday metallographic sample preparation to aid in failure analysis.

2.3.2.13 Universal Hardness Testers. A Rockwell Universal Hardness Tester (figure 2-9), along with several portable hardness testers, is used in determining the hardness of certain alloys used in many special applications.

2.3.2.14 Surface Cleaning Equipment. Surface cleaning equipment, such as the vapor degreaser (figure 2-9), is used in sample surface preparation for electroplating with the large power supply unit. These techniques are used for corrosion studies and edge preservation of metallographic specimens.

2.3.2.15 Scanning Electron Microscope. A scanning electron microscope (figure 2-10) provides direct observation of objects from about 20X to 50,000X, with a resolution approaching 100 angstroms, and all of the field in focus except for lower magnification of very irregular samples. An accessory provides for observing relative conductivity. Photographic stereo pairs, giving a three-dimensional effect for better understanding of the surface, can be made quickly and easily. The instrument is used primarily to give high-resolution, high-magnification surface detail over a relatively large field for electron fractography. However, it can be used to look at virtually any type of material that can withstand the effects of the vacuum and the electron beam, Among types of materials commonly studied with such equipment are all types of metal parts and surfaces, biological specimens, microelectronic circuits and semiconductor devices, plastics and other nonconducting materials. Growth mechanisms and products such as whiskers, corrosion products, and other surface effects due to intentional or accidental handling are also frequently studied. The scanning electron microscope is a unique tool which quickly and easily extends useful vision to the microscopic level over a large field, thus permitting better understanding of the subject.

2.3.3 MECHANICAL AND FLUIDS LABORATORY

2.3.3.1 Pneumatic Console. This console (figure 2-11) provides a low-volume flow output at pressures up to 15,000 psig to facilitate the analysis and test of failed pneumatic components. The console's circuitry is divided into three separate banks: one for low-pressure/vacuum testing from approximately 0.1 psia to 25 psia, the second for pressure testing in the range of 0 to 2,000 psig, and the third in the high-pressure bank reaching 15,000 psig. Test media can be gaseous nitrogen, gaseous helium, air, and gaseous oxygen, utilizing special circuitry included in the console for GOX testing. All valves and regulators are the fine-control type, and the test data gages are accurate to ±0.1 percent of full scale.

2.3.3.2 Hydraulic Console. This hydraulic console (figure 2-12) is capable of producing a pressure output of 3,000 psig at 30-gpm flow rate, or 5,000 psig at a reduced flow rate of 15 gpm. The static test capability is 20,000 psig. A full range of flowmeters is provided to measure fluid flow rates from 200 cc/min to 30 gpm, with excellent readability. A graduated cylinder is used to measure extremely low flow or leakage rates below 200 cc/min. The unit is equipped to test virtually all hydraulic components from the simplest hand valve to engine-driven pumps. The test medium utilized is MIL-H-5606 hydraulic fluid.

2.3.3.3 X-Ray Image Intensifier. This unit (figure 2-13) provides the best means available today for both quick-look and detailed malfunction investigative work. It includes provision for both direct and remote viewing of the test object, in motion or static, and allows for either still or cine photography of X-ray images of test items. The unit handles samples up to 2 feet cubed and 600 pounds in weight; the smallest acceptable component has not been determined; however, a 0.0002-inch-diameter gold wire in a semiconductor has been successfully viewed and photographed. The test sample can be viewed directly on a fluoroscope, or remotely on a vidicon, closed-circuit TV monitor. The vidicon system permits approximately 20 times magnification. Both systems are aided by a movable sample table which can be tilted, rotated, or translated in 3 degrees of freedom. The cine camera, used to film moving parts of components under X-ray, operates at speeds up to 500 frames/sec, and can be slowed down, as required, for analysis purposes.

2.3.3.4 Optical Comparator. The MIL's metrological instruments are headed by a 30-inch optical comparator (figure 2-14) which facilitates viewing and measurement of an object up to 10 inches squared, and up to a magnification of 200 times. Two light sources provide viewing by either the shadowgraph method or by a true image. Measurement readability with this instrument is 0.0001 inch in both the X and Y planes. Other metrological instruments include a toolmaker's microscope, an 18-inch optical comparator, sets of various types of inside and outside micrometers, calipers, gage blocks, hole gages, height gages, and dial indicator sets. Numerous different type and size borescopes and fiber optics devices are similarly available for visual inspection of otherwise inaccessible areas.

2.3.3.5 Infrared Scanner. The Infrared Scanner (figure 2-15) consisting of a Scanning Unit and a Display Unit, detects radiant energy from the object being viewed and translates the thermal energy into electrical energy. The electrical output, combined with the positional information of the vertical and horizontal scanning signals, is applied to the Display Unit where it is presented as a picture of the radiant energy intensity over the surface of the object viewed. A camera system provides instant 4-inch by 5-inch photographs directly from the oscilloscope display.

Two types of display are available. A television type display, called C-scan, permits viewing of the entire surface of the object as a relief map of thermal intensity. Radiation intensity is presented in B-scan type display. In B-scan, the horizontal trace represents distance across the object, and vertical deflection represents radiation intensity. Any line across the object may be selected for analysis; and vertical amplitude may be calibrated, enabling quantitative measurements.

A random selection from the varied applications includes detection of subsurface and fatigue cracks in materials; flaws in metal skins; voids and flaws buried in rubber and plastic structure; flaws and manufacturing errors in multilayer, etched circuit boards; and defective bonds between materials.

2.3.3.6 Laminar Flow Clean Bench. The Laminar Flow Clean Bench (figure 2-16) provides an easily accessible, clean area for visual and microscopic examination of parts and components, and for clean disassembly of small components. Its cleanliness level is the same as that of the clean room, i.e., Class 100.

2.3.3.7 Clean Room. The Clean Room (figure 2-17) is an enclosed area in which the particulate matter in the air, the temperature, the humidity, and the pressure are controlled as required. The Clean Room is designed to provide the environmental control necessary for the disassembly, test, and reassembly of components and assemblies whose operational value would be destroyed if exposed in noncontrolled cleanliness areas.

The MIL's Laminar Flow Clean Room conforms with the definition given in Federal Standard 209 for Class 100 Clean Rooms, which are the highest operational level clean rooms available. It is equipped with ultrasonic cleaners and spray rinses which prepare components for tests and maintain cleanliness of tools, equipment, and apparatus. A pneumatic console is housed in the clean room to provide test pressures up to 2,000 psig, low-flow conditions, utilizing gaseous nitrogen or gaseous helium test media. Two automatic particle counters are available: one for monitoring cleanliness levels in liquids, the second for gases. The latter is used to verify the cleanliness integrity of the clean room itself.

2.3.3.8 Photo Operations Laboratory. The Photo Operations Laboratory (figure 2-18) provides accurate portrayal of anomalous conditions observed in subjects under materials evaluation and malfunction investigation. These records are used as reference information during the investigation and to illustrate findings in the MAB reports.

The laboratory equipment includes a Bausch and Lomb photomicroscopy model L instrument capable of producing high and low magnification photomicrographs illuminated by transmitted, oblique-surface, oblique-transmitted, or any combination of lighting thereof. A light-polarizing device permits the recording of a stress pattern in the subject material.

A Burke and James View Camera, and an MP3 Industrial Polaroid assembly with accessories provide a wide scope of applications including black and white, color, or infrared film data results.

A microscope reflex-view arrangement allows composition of minute subject matter for illustrating discrepancies. Photo laboratory support equipment such as densitometers, fibre optics, and lighting accessories augment operations.

2.3.3.9 Dissection and Sample Preparation Facility. This facility is equipped with a precision milling machine and two lathes capable of disassembling components,

including those irregularly shaped. In addition, both the milling machine and the two lathes can be used for making precision measurements such as concentricity and runout. Other equipment available includes a variable-speed drill press, a band saw, a cut-off saw, a belt sander, and grinders. This equipment is used in conjunction with the milling machine and the two lathes for dissection and sample preparation required in the course of materials evaluations and malfunction investigations. In addition, this facility is used for the preparation of metallic chemical samples for the MAB, Support Laboratory (SL).

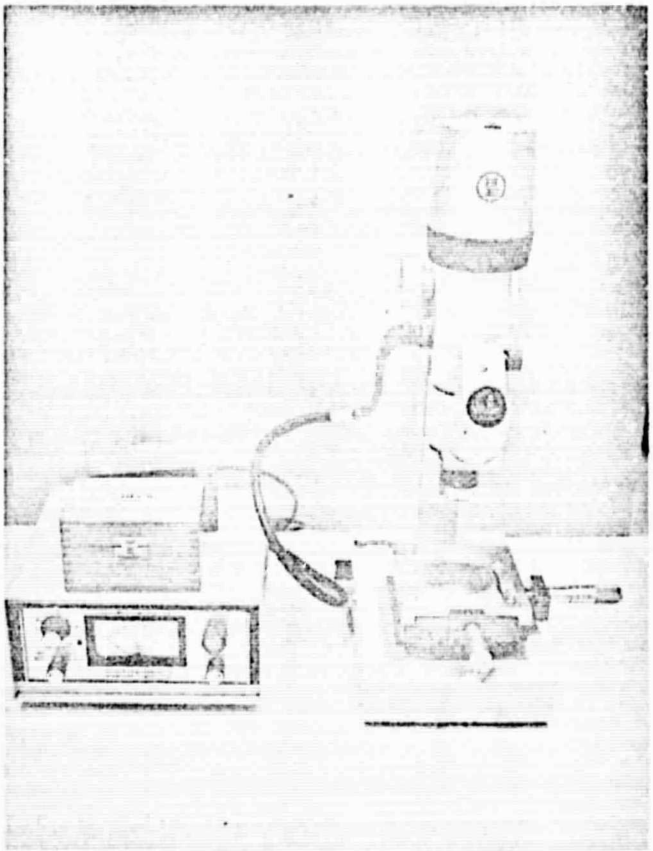

Figure 2-1. Infrared Microscope

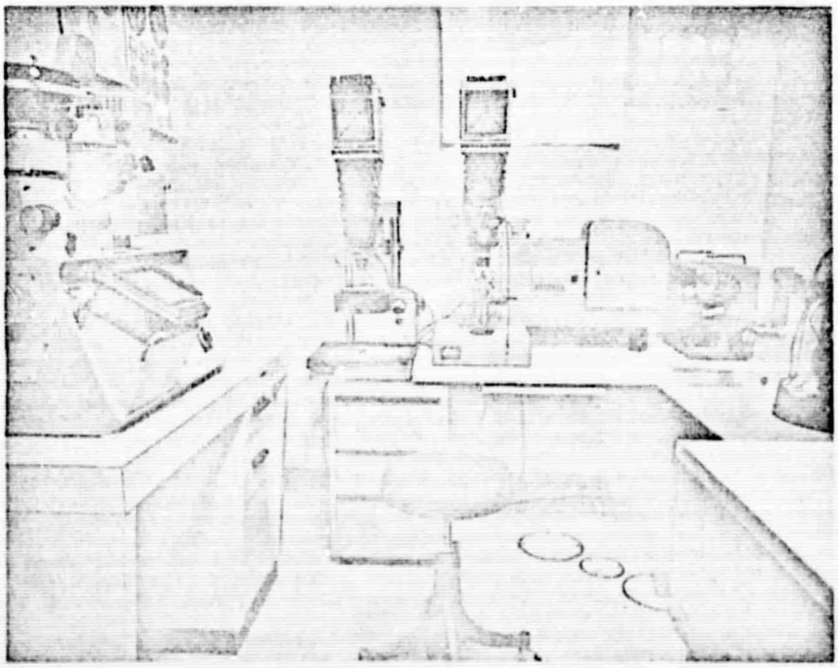

Figure 2-2. Optical Metallography Equipment (from left to right) Vickers 55 Metallograph, Leitz Wetzler Aristophot Macro Camera, Leitz Wetzler Panphot Metallograph, and a Low-Power Binocular Stereo Microscope

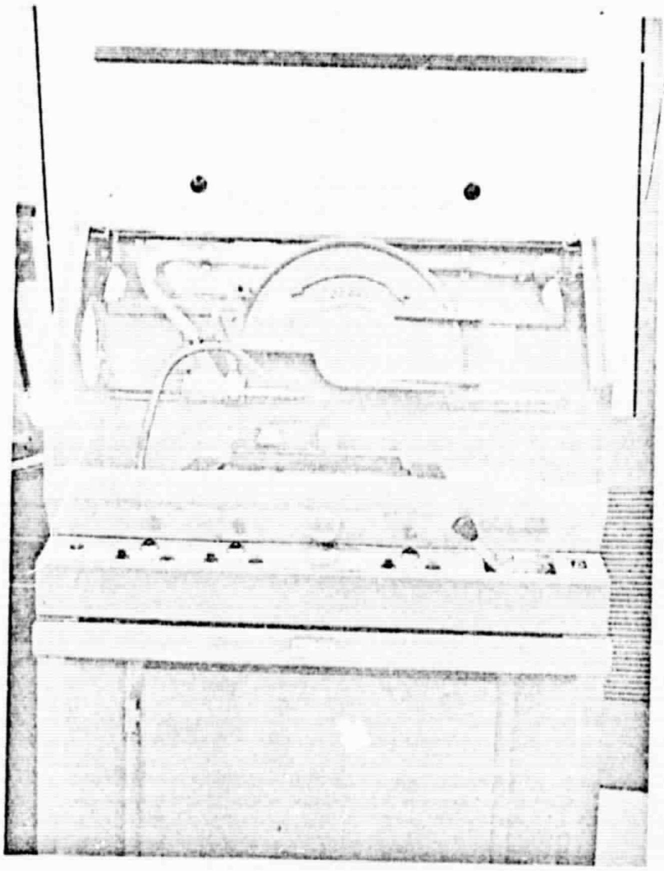

Figure 2-3. Cut-Off Saw

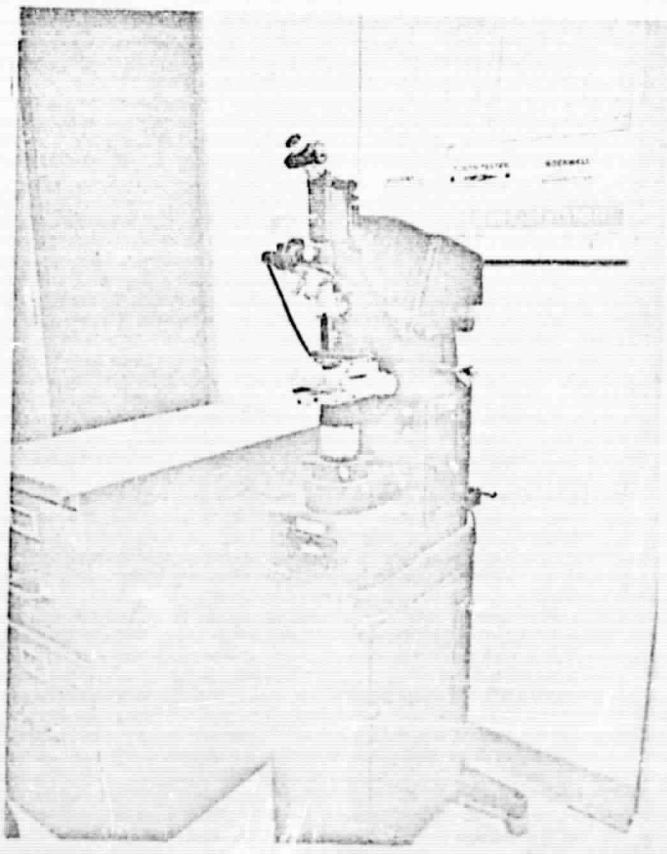

Figure 2-4. Tukon Microhardness Tester

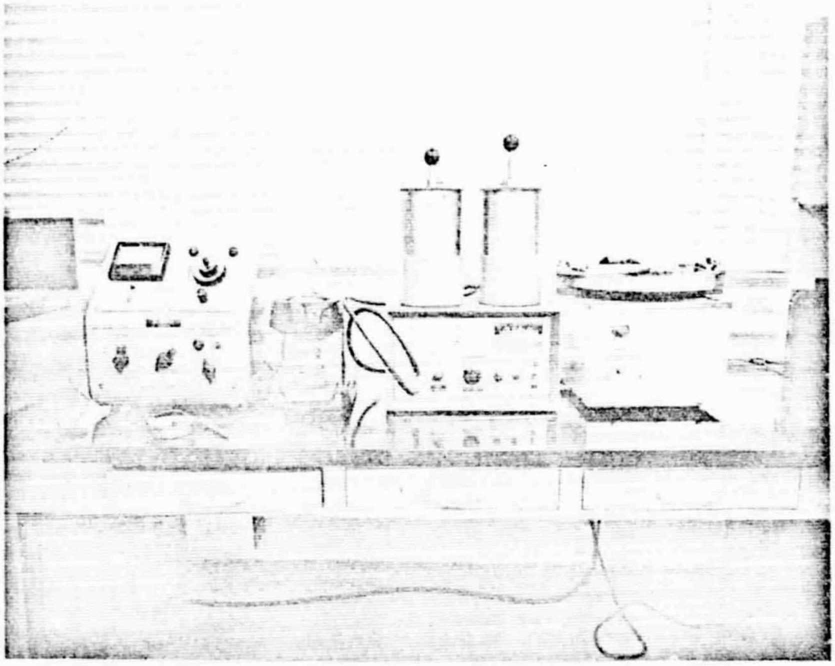

Figure 2-5. Electrolytic Etcher, Ultrasonic Cleaner, and Automatic Vibrating Polisher (depicted left to right)

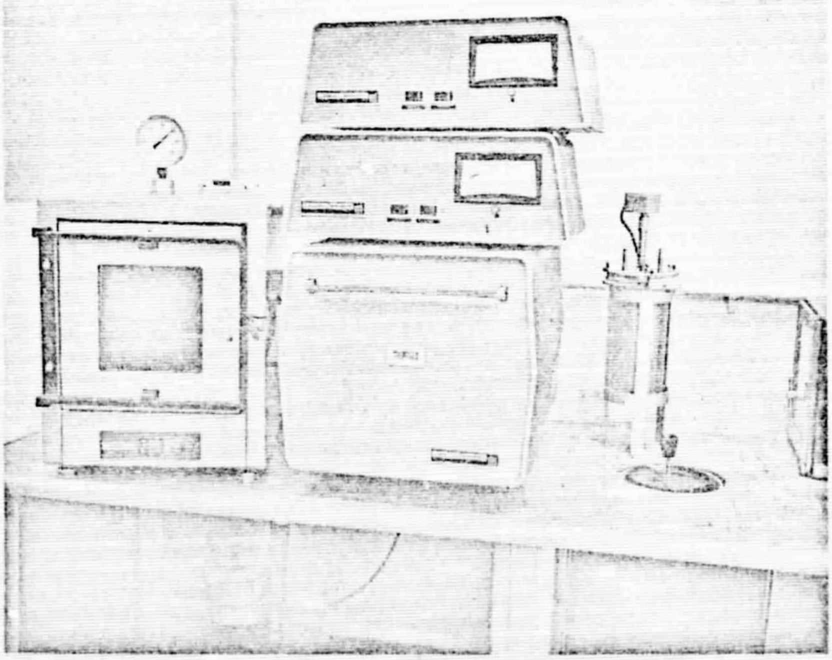

Figure 2-6. Heat Treating Furnaces

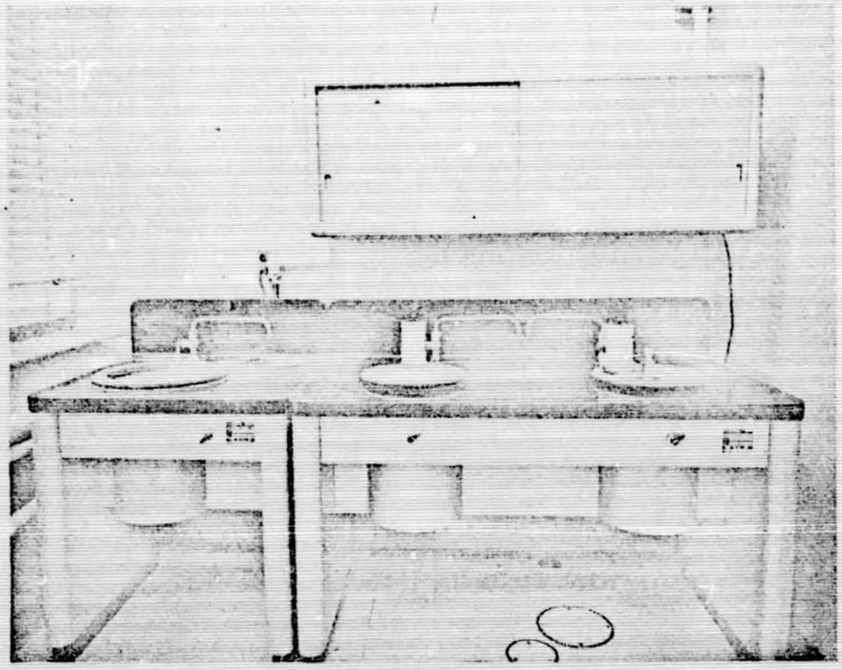

Figure 2-7. Lapping Wheels

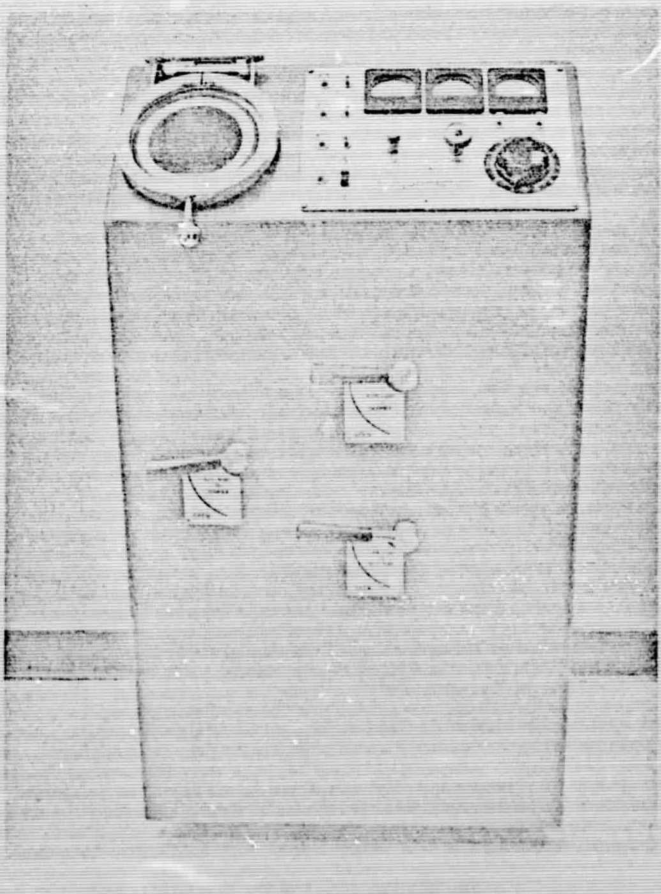

Figure 2-8. Vacuum Cathodic Etcher

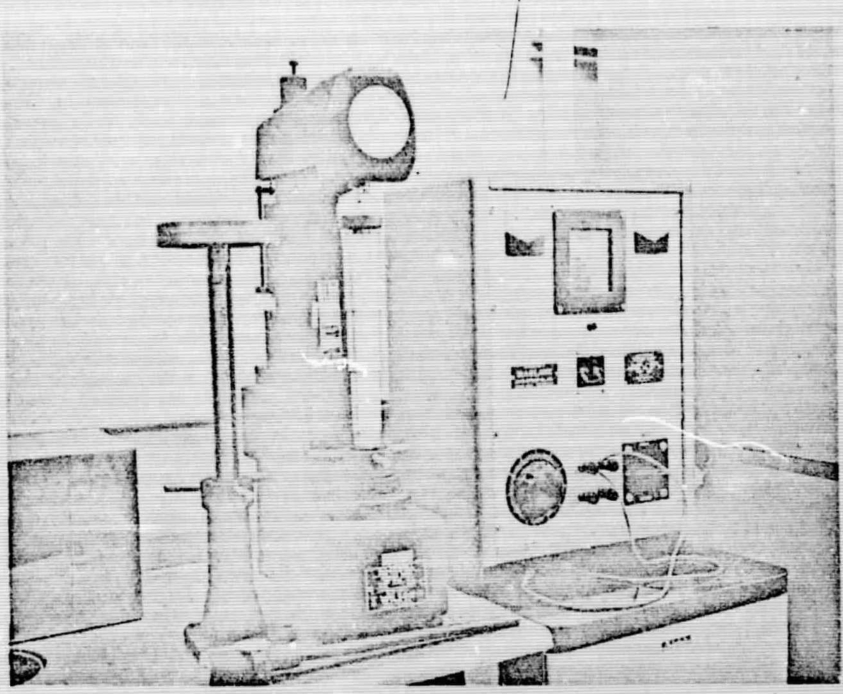

Figure 2-9. Universal Hardness Tester and Electroplating Power Supply (right)

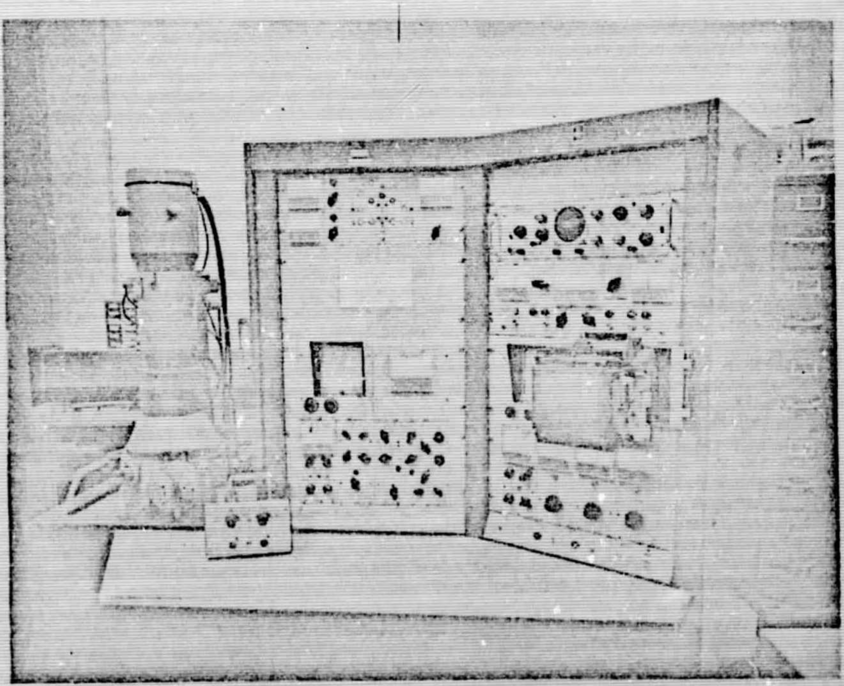

Figure 2-10. Scanning Electron Microscope

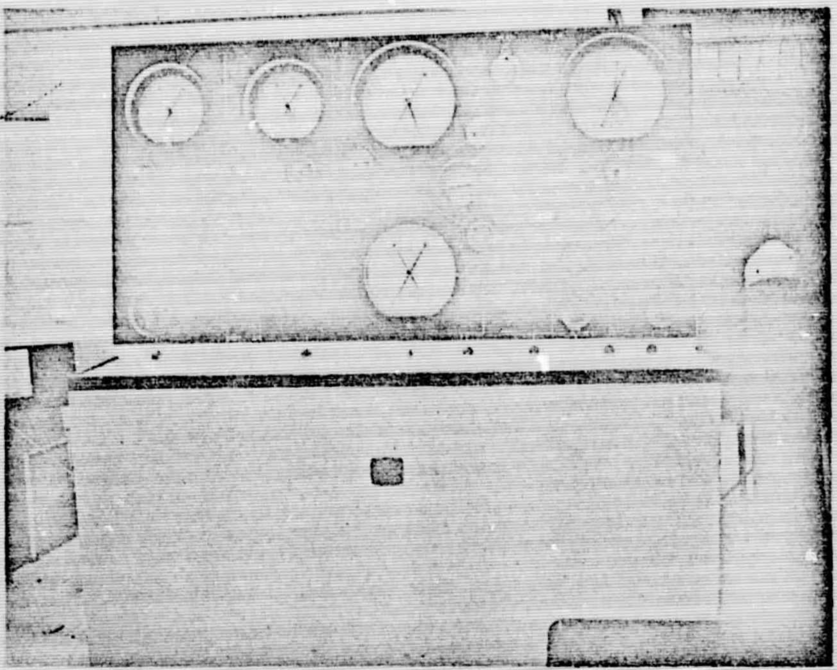

Figure 2-11. Pneumatic Console

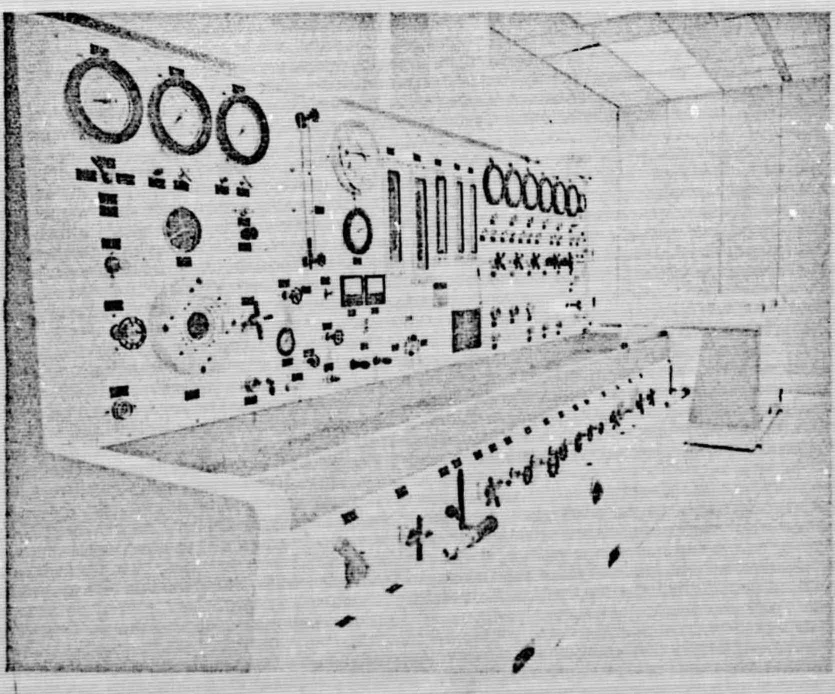

Figure 2-12. Hydraulic Console

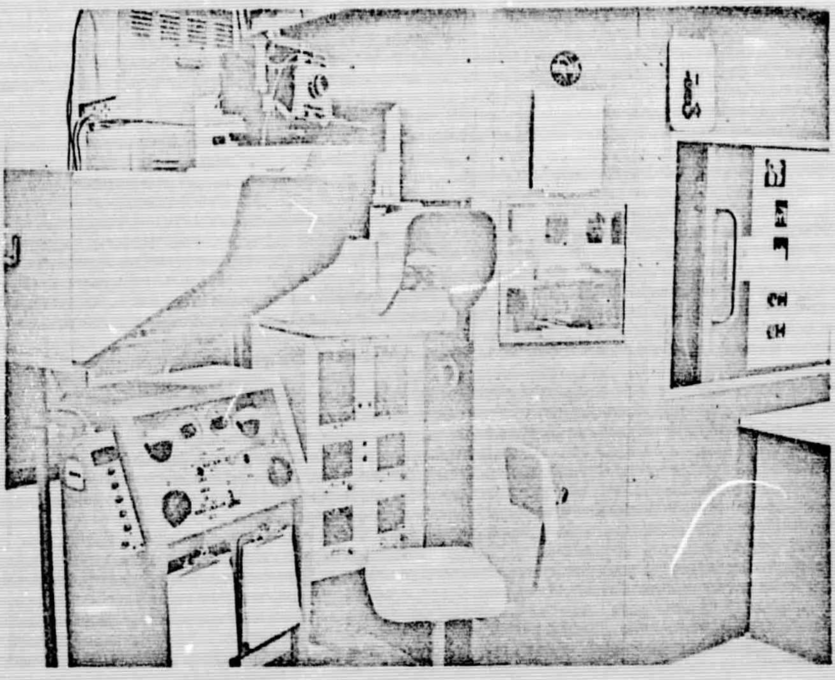

Figure 2-13. X-Ray Image Intensifier

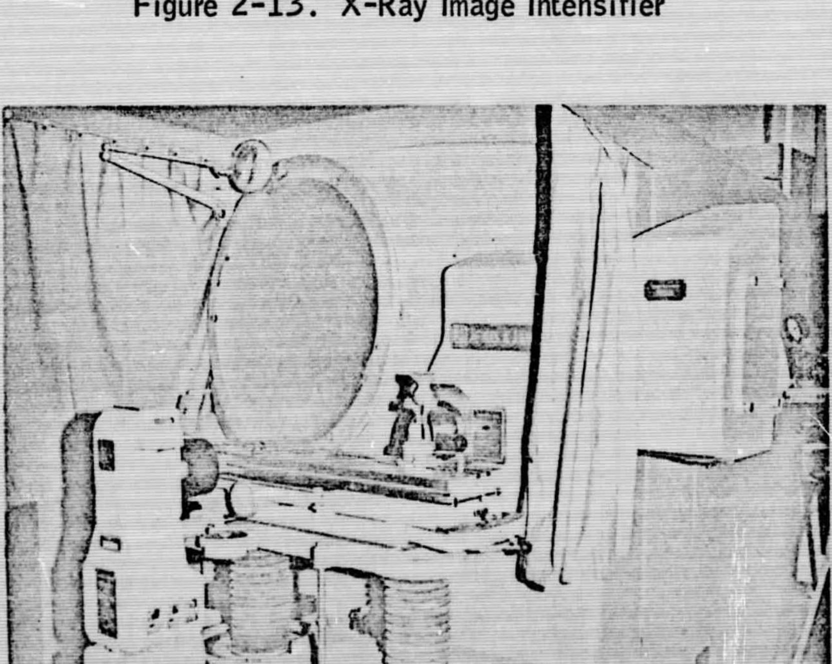

Figure 2-14. Optical Comparator

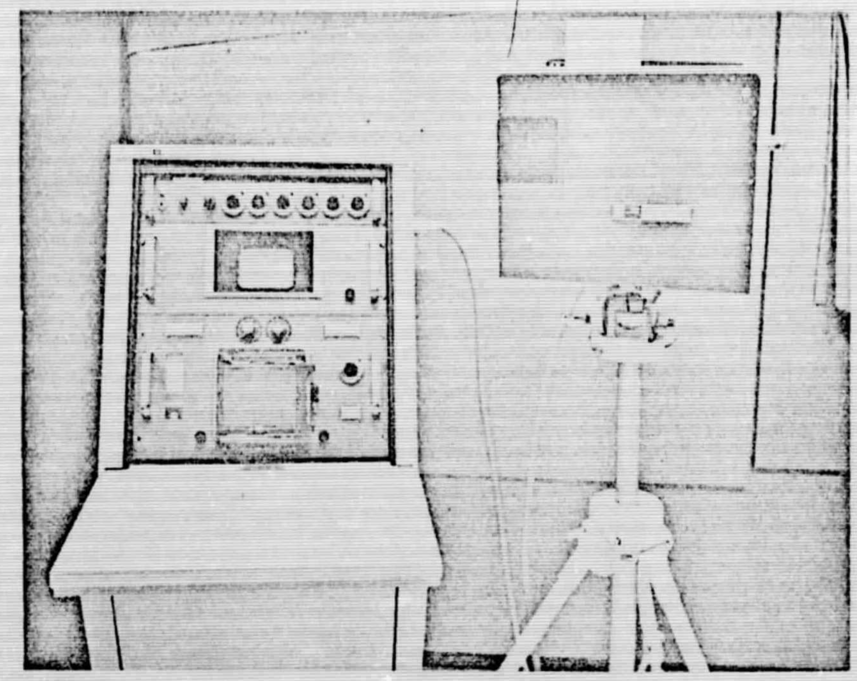

Figure 2-15. Infrared Scanner

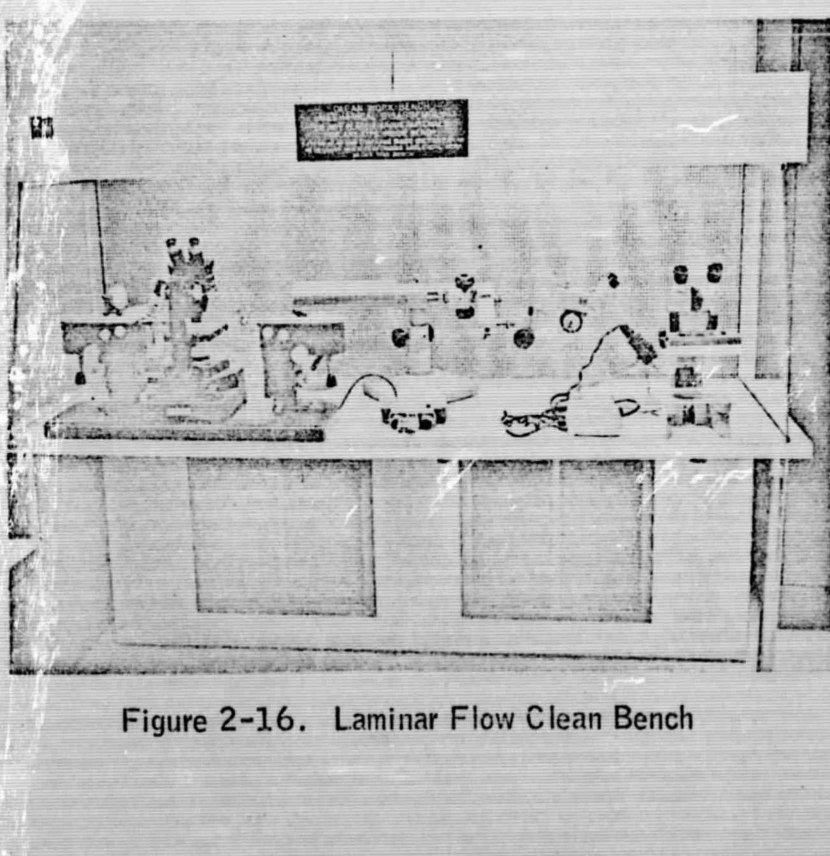

Figure 2-16. Laminar Flow Clean Bench

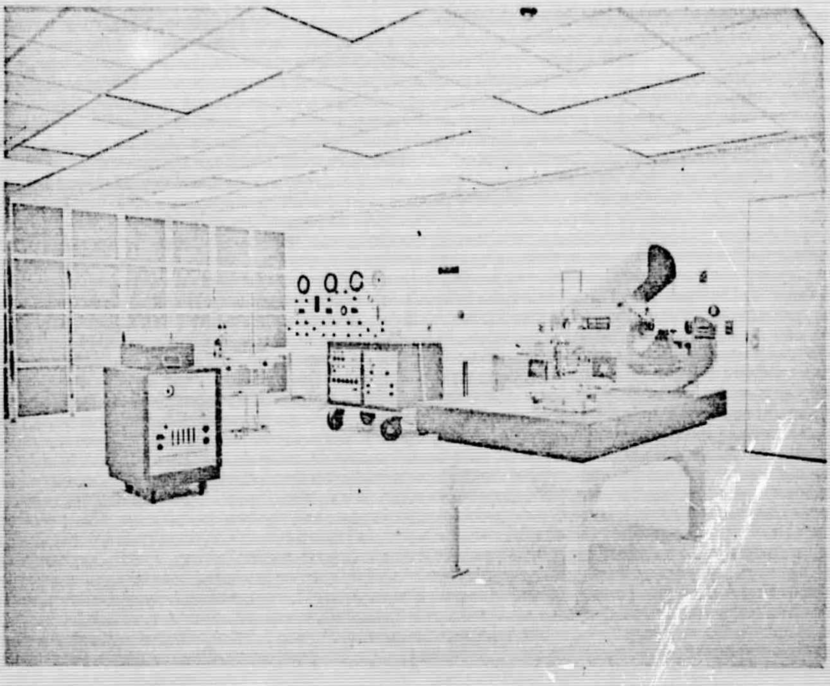

Figure 2-17. Clean Room, Class 100

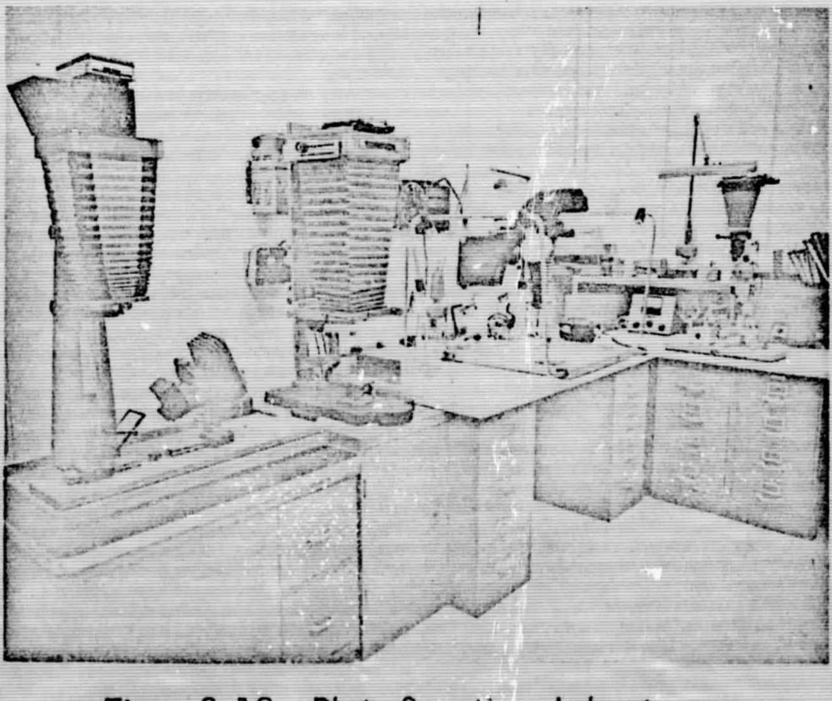

Figure 2-18. Photo Operations Laboratory

SECTION III
FACILITIES AND EQUIPMENT
SUPPORT LABORATORY SECTION

3.1 GENERAL

The Support Laboratory Section has the capability of performing almost any chemical analysis by either classical or state-of-the-art instrumental methods. The laboratory has particularly specialized in the analysis of microscopic samples and is equipped to provide this service for rapid response to operational problems. The services of the laboratory and its staff of 11 professional chemists are used by the Center for the following tasks.

3.1.1 MALFUNCTION INVESTIGATION

Chemical analyses for malfunction investigation, like forensic methods, require very close cooperation between chemist and investigator. The Support Laboratory Section and the Malfunction Investigation Laboratory Section at Kennedy Space Center are located in the same building for easy consultation and to facilitate the removal and handling of microscopic samples. Such samples typically require analysis times of a few hours to a few days and demand the highest level of professional competence from the laboratory.

3.1.2 GENERAL NONROUTINE ANALYSIS

The laboratory is equipped to analyze samples for major components, trace components, elemental composition, and surface composition. Extensive facilities are available for analysis of organic materials and determination of molecular structure by infrared and mass spectroscopic methods.

3.1.3 METHODS DEVELOPMENT AND SPECIALIZED ANALYSIS

Standard analytical methods are frequently not available to meet the unexpected problems that occur during launch preparation and checkout. Under such conditions, the

Support Laboratory is in a position to simultaneously draw on the knowledge of a team of specialized analytical chemists, search the chemical literature, apply the latest techniques in analytical instrumentation, and coordinate the results with the cognizant systems engineers. These same specialists serve as an analytical development group supplying new methods for routine use by other laboratories.

3.1.4 CHEMICAL RESEARCH AND DEVELOPMENT

The scope of laboratory work has recently been expanded to include short-term chemical research and development. Capabilities in this area would include chemical kinetics (e.g., corrosion rates), decomposition mechanisms, contamination studies, etc. Detailed studies of materials can be made to determine the nature and extent of material degradation under almost any thermal or chemical environment.

3.2 REFERENCE FACILITIES

Effective work in the many chemical disciplines outlined above requires extensive library and reference facilities. Some of these reference facilities are listed below:

a. In keeping with the philosophy of maintaining a total capability in analytical chemistry, the laboratory maintains a complete file of Sadtler reference spectra (approximately 100,000 spectra), and Sadtler thermograms. These spectra are complemented by "Coblentz Society Spectra" as well as many smaller publications.

b. Complete ASTM data files for X-ray diffraction (15,000 compounds) and mass spectroscopy are also in the laboratory, as well as the other commercially available sources in these fields.

c. In addition, the Center library maintains major reference collections such as Chemical Abstracts, Beilstein, and many current periodicals for laboratory use.

d. The laboratory also maintains an extensive collection of standard, samples (particularly alloys) from the National Bureau of Standards, British Bureau of Analyzed Samples, and other sources. These materials are carefully analyzed and have certified compositions. Within the laboratory, efforts are constantly under way to provide good secondary standards by analysis and checking of commercially available alloys, for which

primary standards are not available. There is also an active program to provide new reference data for the identification of organics by infrared and mass spectroscopy and by X-ray diffraction.

e. The laboratory has a stock of well over 1,000 different chemicals that may be used as uncertified standards.

3.3 WET CHEMICAL LABORATORY FACILITIES

The laboratory has a physical plant suitable for handling, synthesis, purification, and analysis of materials ranging from hypergolic propellants to small quantities of explosives and highly toxic chemicals. The wet chemistry laboratory utilizes a major portion of this bench space and has apparatus for most classical methods of analysis.

The Wet Chemistry Laboratory also has equipment (some of which is shown in figure 3-1) for the following:

- Atomic Absorption Spectrophotometry
- Flame Emission Spectroscopy
- Polarographic Analysis
- C, H, N determination
- Ultraviolet-Visible-Near Infrared Spectrophotometry
- Electrodeposition
- Conductometric, Potentiometric and Colorometric Titrations
- Carbon and Sulfur in metals
- Purification and Separation by techniques such as Distillation, Extraction, and Thin-Layer Chromatography

3.4 INSTRUMENTAL ANALYSIS FACILITIES

3.4.1 INFRARED SPECTROSCOPY

The infrared spectroscopy laboratory has two Perkin-Elmer Model 621 spectrophotometers (figure 3-2), each with 6X front surface beam condenser; and a Perkin-Elmer Model 221 spectrophotometer with 8X beam condenser. Accessories include equipment for KBr work, reflectance, and polarization; as well as cells for solids, liquids, and gases.

3.4.2 X-RAY FLUORESCENCE AND DIFFRACTION

The X-ray laboratory has facilities for X-ray fluorescence (Norelco spectrograph) and three diffraction generators (figure 3-3) to permit X-ray analysis by a variety of methods. Accessories to the above equipment include a diffractometer with high-temperature capability, pulse height analyzer, and several kinds of diffraction cameras.

3.4.3 DIFFERENTIAL THERMAL ANALYSES

The thermal analysis laboratory has a Mettler thermal analyzer (figure 3-4). This instrument is equipped to simultaneously record differential changes in weight and temperature of a sample, while the sample temperature is varied from room temperature to 3,000° F. Samples can be run, under these conditions, in various controlled atmospheres or under hard vacuum. Provisions can also be made for off-gas analysis.

3.4.4 GAS CHROMATOGRAPHY

The gas chromatography laboratory has four chromatographs. These instruments (figure 3-5, typical) provide a capability for analytical and preparative chromatography. Sampling systems are available for solids, liquids, gases, sealed containers, high pressures, and pyrolysis. Available detectors include flame, thermionic, electron capture, thermal conductivity, and micro cross-section.

3.4.5 EMISSION SPECTROSCOPY

The emission spectroscopy laboratory has Jarrell-Ash 3.4 meter (Ebert) and 1.5 meter (Wadsworth) spectrographs (figure 3-6) and a Jarrell-Ash recording densitometer. The Ebert spectrograph has an arc stand and varisource source while the Wadsworth is equipped with a laser microprobe.

3.4.6 MASS SPECTROMETRY

The laboratory has two mass spectrometers: a Consolidated Electrodynamics Corporation (C.E.C.) Model 104 with gas chromatograph interface (figure 3-7), and a C.E.C. Model 110 double-focusing instrument (figure 3-8). Both instruments are equipped

with direct-introduction systems for solid samples as well as conventional inlet systems. The Model 110 can be converted to an rf spark source instrument for solids by changing sources.

3.4.7 ELECTRON MICROSCOPY

An electron microscope, Hitachi Model HU-11A, (figure 3-9) can provide direct magnifications to approximately 300,000X. This can be improved photographically to about 1,500,000X. The instrument also has facilities for high-resolution electron diffraction and stereo photography. An LKB ultra microtome, shadowing equipment, and various sample stages are also available.

3.4.8 ELECTRON MICRO X-RAY ANALYSIS

The laboratory's electron micro X-ray analyzer is an Applied Research Laboratories Model EMX (figure 3-10). This instrument is equipped for light-element work and has two pulse height analyzers, three scalers, a four-channel recorder, two oscilloscopes, and magnetic sweep drive. Plans are presently under way to install a lithium-drifted silicon nondispersive detector.

3.4.9 ELECTRON SPECTROMETRY

The laboratory has recently purchased an electron spectrometer for chemical analysis (ESCA). This instrument will be used for surface analysis and molecular structure determination.

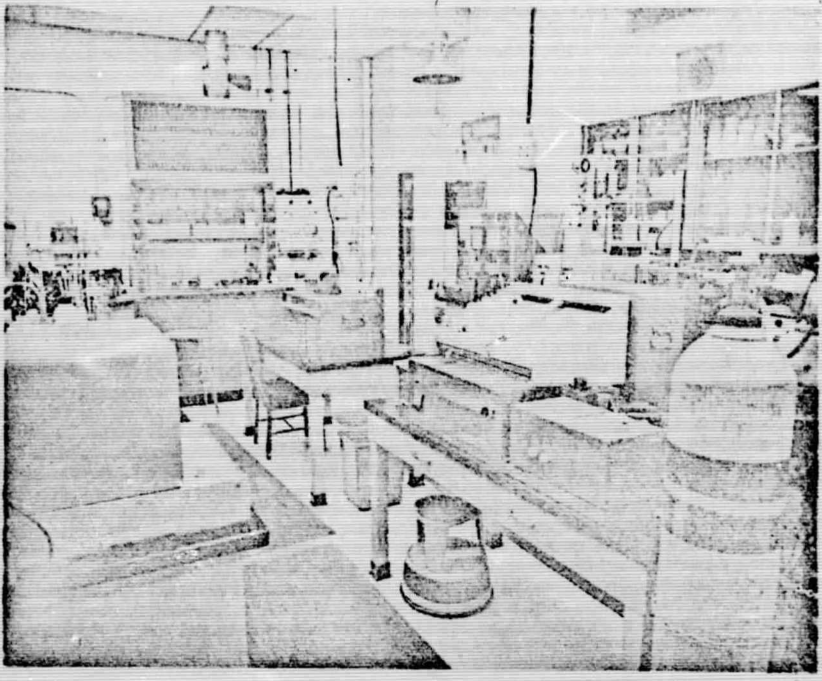

Figure 3-1. Portion of Wet Chemistry Laboratory

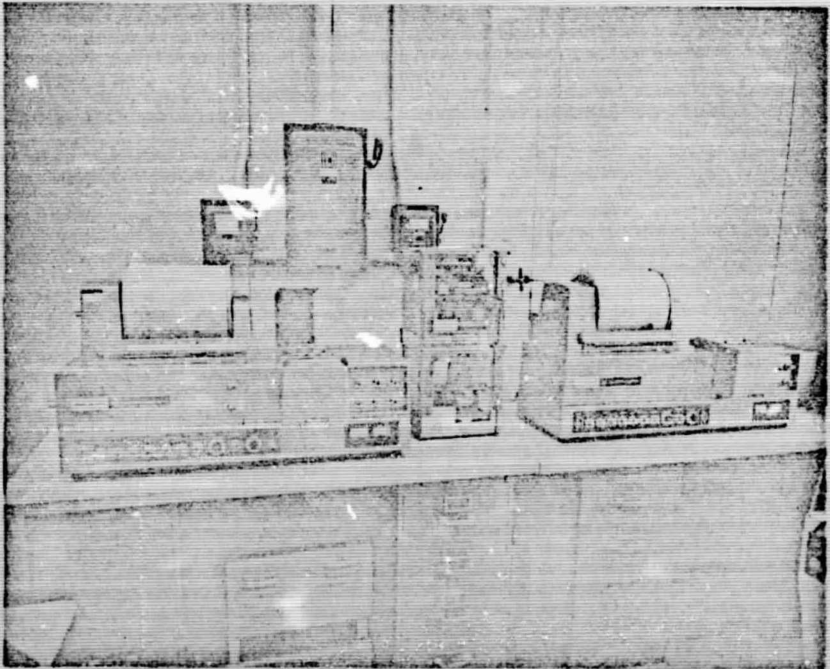

Figure 3-2. Infrared Spectrophotometers

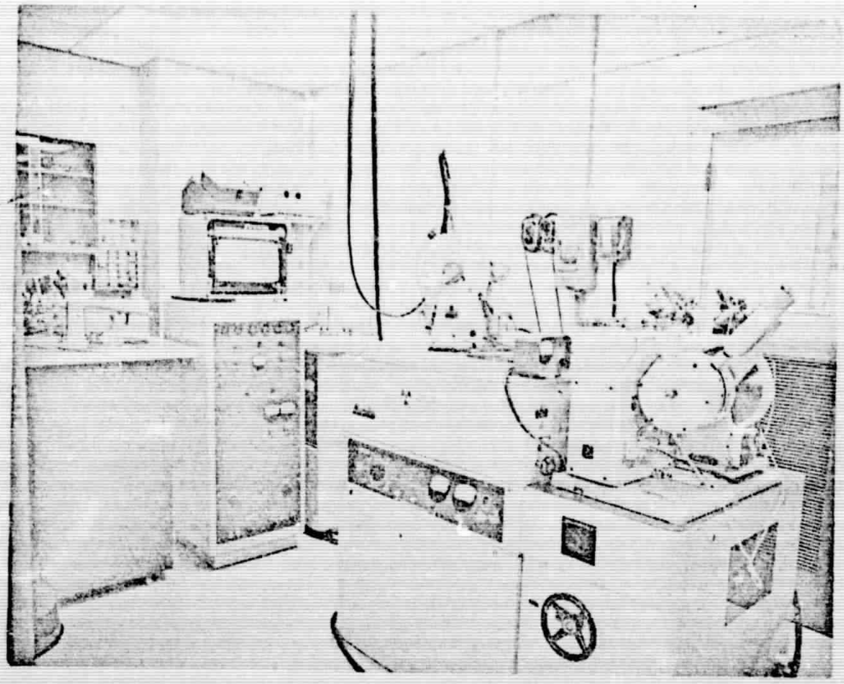

Figure 3-3. X-Ray Fluorescence and Diffraction Generators

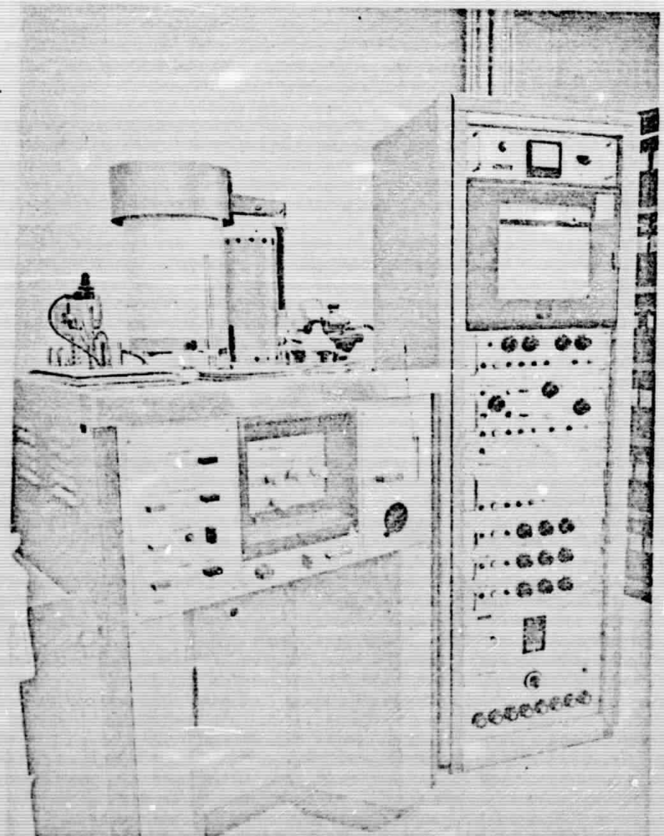

Figure 3-4. Differential Thermal Analyzer

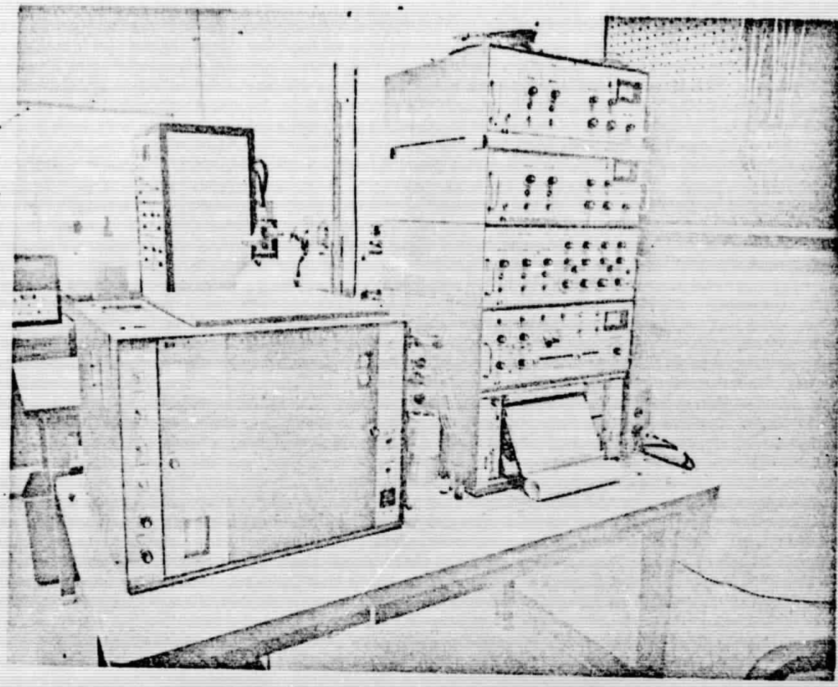

Figure 3-5. Gas Chromatographs

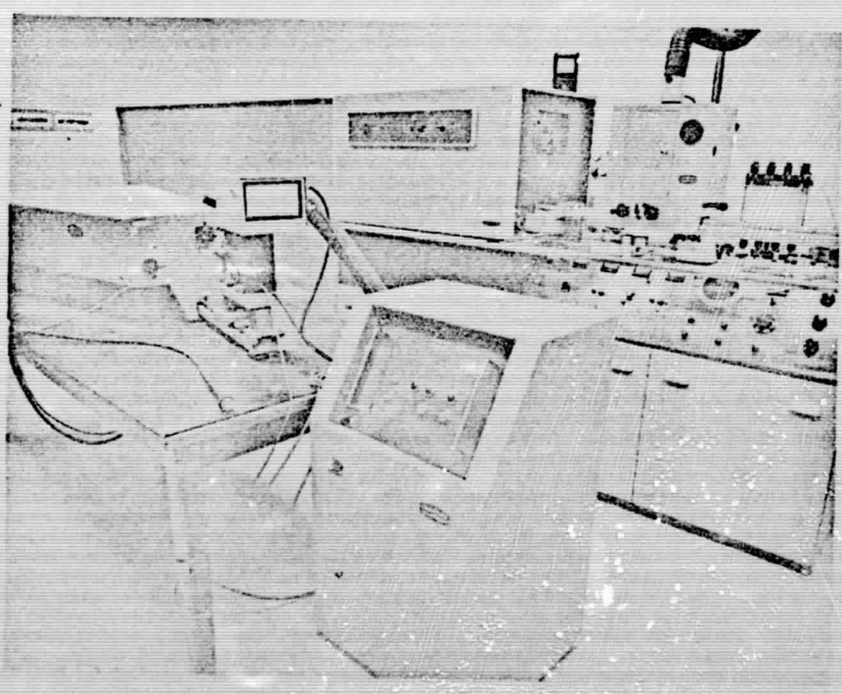

Figure 3-6. Emission Spectrographs

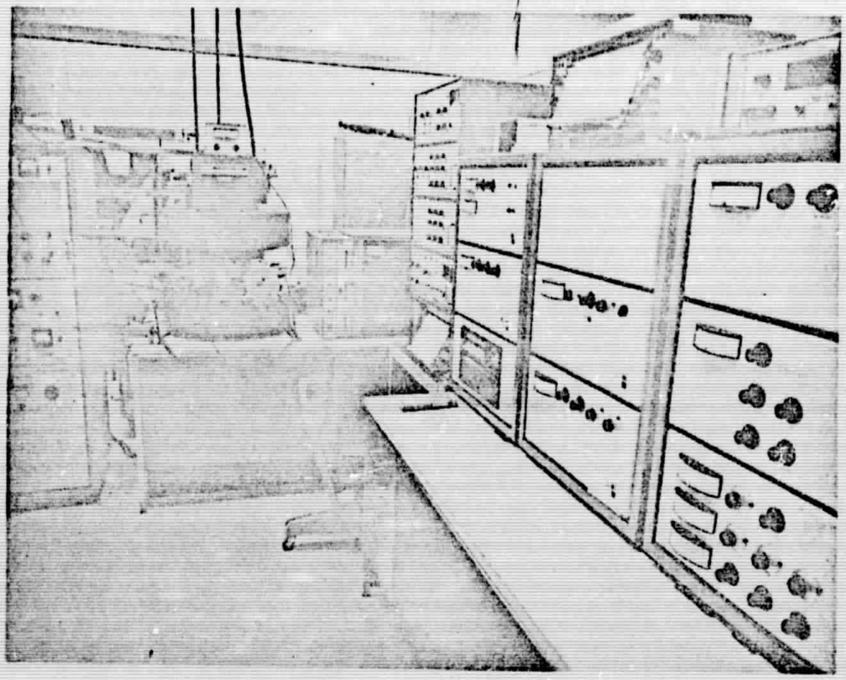

Figure 3-7. Mass Spectrometer, Model 104

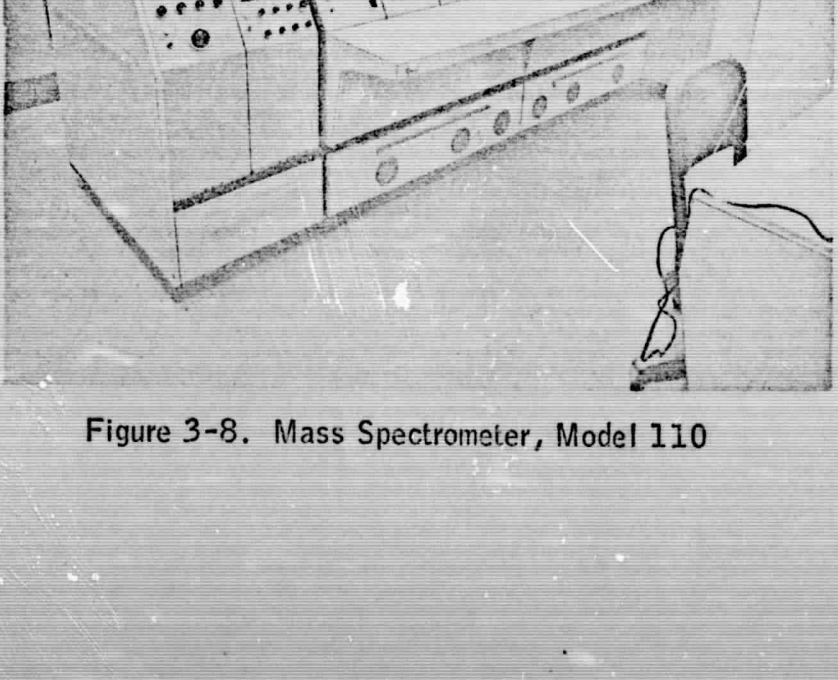

Figure 3-8. Mass Spectrometer, Model 110

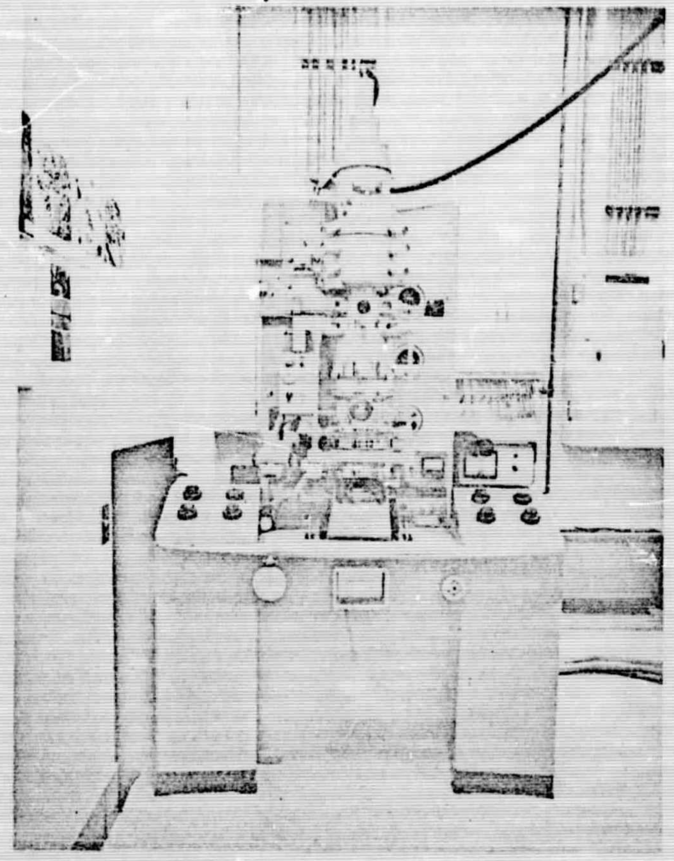

Figure 3-9. Electron Microscope

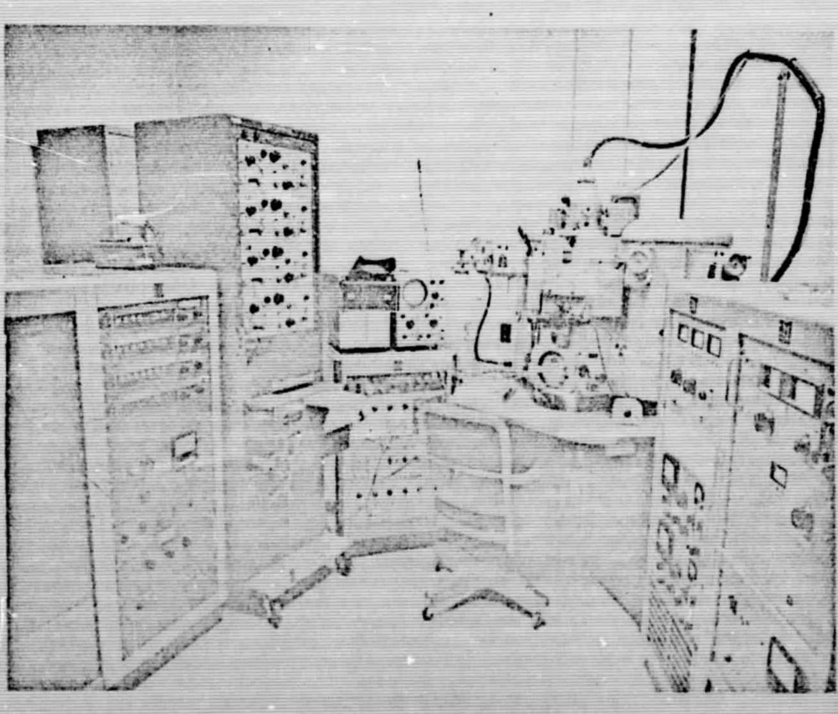

Figure 3-10. Electron Micro X-Ray Analyzer

APPROVAL

GP-225, Rev. 1

MALFUNCTION INVESTIGATION
AND
CHEMICAL ANALYSIS
CAPABILITY MANUAL

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